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Removal of Polynuclear Aromatic Hydrocarbons From Primary Aluminum Air Pollution Control Scrubber Wastewater

Rexnord, Inc., Milwaukee, WI

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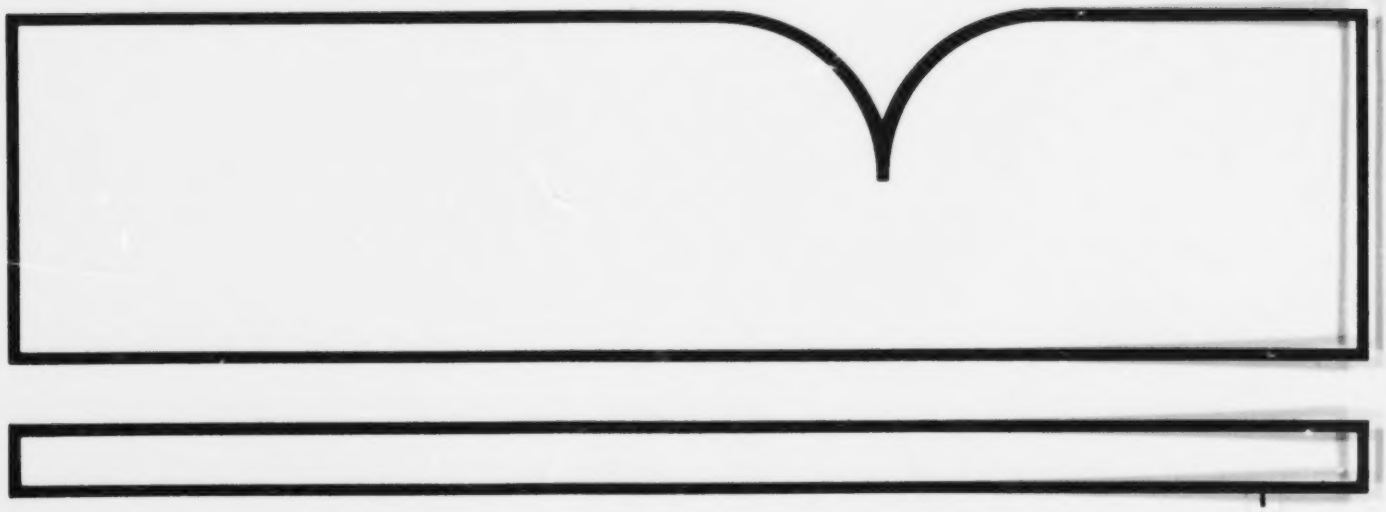

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REMOVAL OF POLYNUCLEAR AROMATIC HYDROCARBONS FROM PRIMARY ALUMINUM AIR POLLUTION CONTROL SCRUBBER WASTEWATER

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16. ABSTRACT

A pilot-scale study was conducted at a primary aluminum plant to evaluate the removal of benzo(a)pyrene and other polynuclear aromatic hydrocarbons (PAH's) from potline scrubber wastewater. Specific objectives included determining the need for granular activated carbon to remove the PAH's to 10 µg/l and evaluating the use of benzo(a)pyrene as an indicator for the removal of all PAH's.

The pilot-scale system removed all PAH's to less than 10 µg/l. For the five major PAH's which ranged from 600 to 3,670 µg/l in the raw waste, 94 to 98 percent removal was achieved by the clarification process. Twenty-seven to 50 percent of the remaining PAH's was achieved by the dual-media filter, and the granular activated carbon columns removed all remaining PAH's to less than 10 µg/l. Benzo(a)pyrene was effectively removed to 10 µg/l with only clarification and filtration. However, granular activatd carbon adsorption was required to remove fluoranthene and pyrene to 10 µg/l. Benzo(a)pyrene was not a good indicator for the removal of all PAH's.

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INTRODUCTION

The primary aluminum smelting industry, standard industrial classification (SIC) 3334, is defined as that industry which produces aluminum metal by the electrolytic reduction of purified aluminum oxide (alumina). All of the aluminum reduction plants in the United States utilize the same process which is known as the Hall-Heroutt process and therefore have very similar wastewaters. The major differences between plants is the manner in which the anode is replaced in the electrolytic cell (1).

The electrolytic reduction of alumina produces large volumes of gaseous products which must be cleaned before release. In addition, the manufacture of pre-baked anodes creates contaminated air streams which must be cleaned. Wet scrubbing is frequently used to control these air streams and represents the major source of wastewater from this industry. This wastewater is known to contain polynuclear aromatic hydrocarbons (PAH's), some of which have been tentatively classified as known or suspected, human or mammalian carcinogens (2).

In March, 1983, the EPA proposed effluent standards (1) which, among other things, limited the release of PAH's from the primary aluminum industry based on the transfer of precipitation and activated carbon technology from other industrial categories. The control of PAH's was based on an achievable concentration of 10 ug/1 of benzo(a)pyrene as an indicator for the removal of all PAH's to 10 ug/1.

During July 1983, a pilot plant study was conducted at a primary aluminum plant to help evaluate the ability of these technologies to remove benzo(a) pyrene and other PAH's from actual primary aluminum potline scrubber wastewater. Specific objectives included determining the need for granular activated carbon to remove the PAH's to 10 ug/1 and evaluating the use of benzo(a)pyrene as an indicator for the removal of all PAH's. A potline wastewater sidestream was treated continuously in a trailer mounted pilot plant for a nine day period. (This test was limited to a nine day period.) The equipment contained in this vehicle is illustrated in Figure 1.

The pilot treatment train consisted of chemical coagulation with ferric chloride and lime; pH adjustment with HCl; cationic polymer addition; clarification; pH adjustment with HCl; dual-media filtration and granular activated carbon adsorption. Figure 2 schematically presents the treatment technology arrangement. The target flow rates were 30 1/minute (8 gpm) for the clarifier; 11.4 1/minute (3 gpm) for the dual-media filter and 9.5 1/minute (2.5 gpm) for the granular activated carbon columns.

In addition, bench-top studies were conducted with this potline scrubber wastewater and with an anode bake scrubber wastewater from another primary aluminum plant to determine if matrix interferences existed.

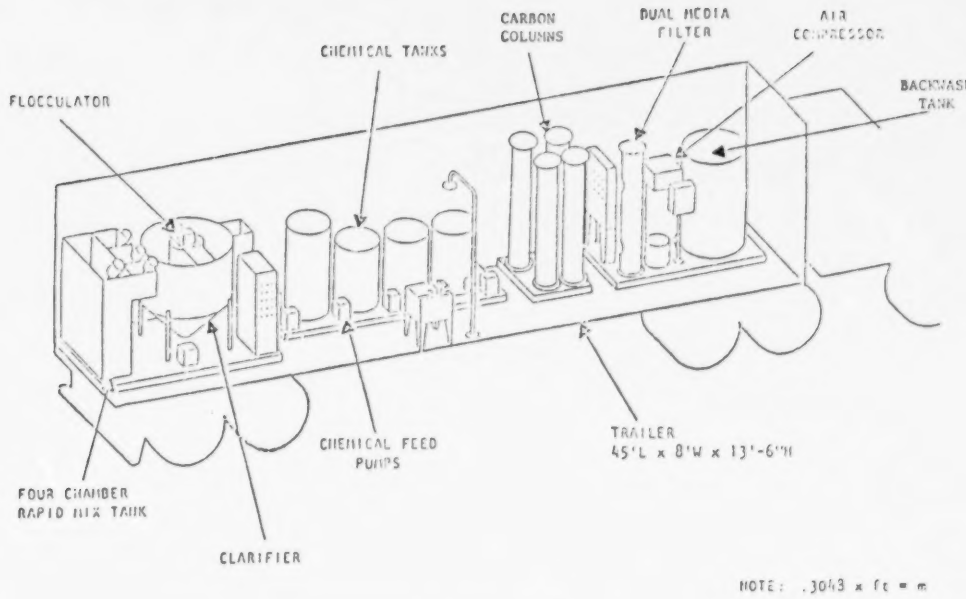

Figure 1. Mobile Wastewater Treatment System

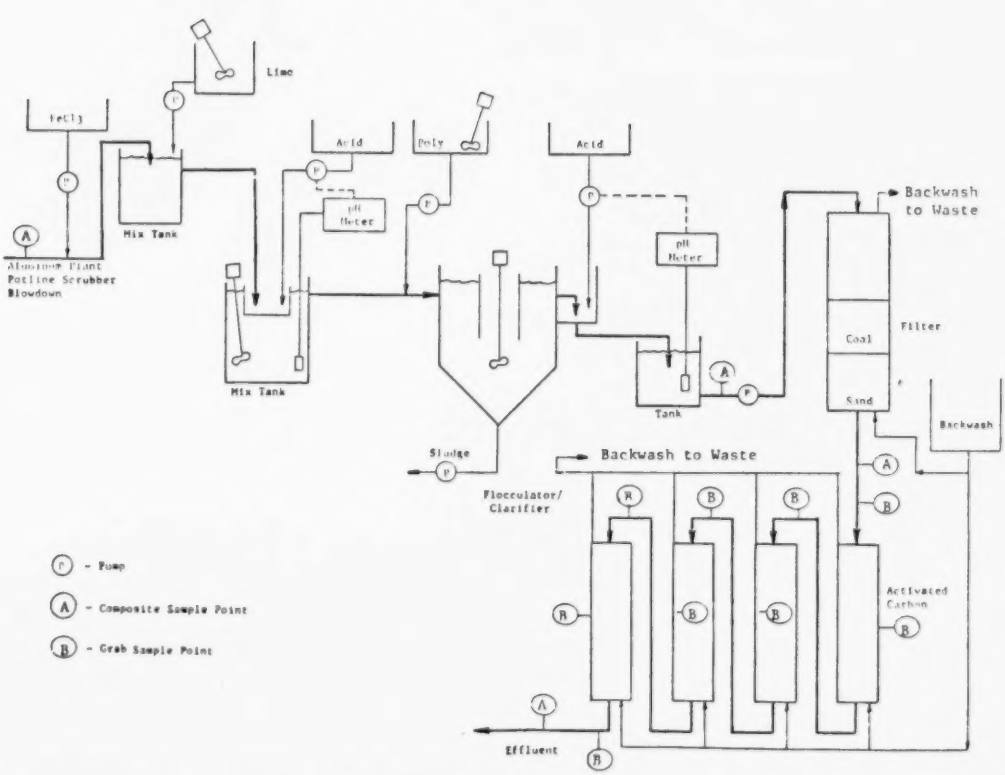

Figure 2. Pilot Treatment Train Investigated
(Aluminum Plant - Potline Scrubber Blowdown)

PILOT PLANT RESULTS

Sample Collection/Analysis

Automatic composite samples were collected daily over 24 hours using EPA Protocol (3) from the following locations:

Raw blowdown
Clarifier effluent
Filter effluent
Activated carbon effluent

In addition, daily grab samples were taken of the feed to the carbon columns and the midpoints and effluents from each column. In accordance with protocol procedure, blank water was pumped through the samplers and analyzed prior to actual data collection. All of the results from the PAH analyses performed on the blank samples were reported as "not detectable".

Laboratory analysis on the composite samples was by EPA Method 625 (2). Selected grab samples from the activated carbon columns were analyzed using EPA Method 610 (2). Some samples were also split and sent to the Industrial Environmental Research Laboratory (IERL-Ci), U.S. Environmental Protection Agency, Cincinnati, Ohio, where they were analyzed for benzo(a) pyrene, fluoranthene and pyrene using Method 610.

Feedwater Quality

Nine composite samples were collected for PAH analysis of the aluminum plant potline scrubber blowdown. The testing results are summarized in Table 1. As shown in Table 1, napthalene, acenaphthylene, fluorene, phenanthrene/anthracene and indeno (1,2,3-cd) pyrene were not detected in the raw waste. Acenaphthene averaged 30 ug/l in the raw waste and ranged from 20-40 ug/l. Fluoranthene, pyrene and chrysene/benz(a)anthracene were the most concentrated PAH's found in the aluminum plant blowdown averaging 2700, 2000 and

2200 ug/1, respectively. The concentration of 3,4-benzofluoranthene/benzo(k) fluoranthene in the raw waste averaged 790 ug/1 for the nine samples tested and ranged from 600-1100 ug/1. Benzo(a)pyrene concentration in the potline scrubber blowdown averaged 1100 ug/1 with a range from 700-1800 ug/1. Dibenzo(a,h) anthracene and benzo(ghi)perylene were present in the blowdown stream in average concentrations of 140 and 310 ug/1, respectively.

The average suspended solids concentration was 130 mg/1 and ranged from 75-238 mg/1.

TABLE 1.
CONCENTRATION OF PAH'S
IN RAW WASTEWATER

PARAMETER	AVERAGE RAW WASTE VALUE	RANGE OF VALUES
1. Naphthalene	ND	ND
2. Acenaphthylene	ND	ND
3. Acenaphthene	30	20 - 40
4. Fluorene	ND	ND
5. Phenanthrene/Anthracene	ND	ND
6. Fluoranthene	2700	1800 - 3700
7. Pyrene	2000	1400 - 2900
8. Chrysene/Benz(a)anthracene	2200	1800 - 3200
9. 3,4-Benzofluoranthene/ Benzo(k)fluoranthene	790	600 - 1100
10. Benzo(a)pyrene	1100	700 - 1800
11. Dibenzo(a,h)anthracene	140	90 - 200
12. Indeno(1,2,3-cd)pyrene	ND	ND
13. Benzo(ghi)perylene	310	220 - 440

ND = not detected (quantification limit = 10 ug/1).

Average was derived from nine analyses

All values reported as ug/1 without correction for recovery.

Analysis by Method 625.

Chemical Clarification

The primary purpose of clarification in this study was to remove suspended solids and associated PAH's present in the aluminum plant potline scrubber wastewater.

Raw potline scrubber blowdown was fed to the Mobile Wastewater Treatment System (MWTS) at a flow rate of 26 - 30 l/min (7-8 gpm). The average ferric chloride dosage was 3180 mg/l which was added in-line. After about 2 minutes of in-line mixing between the ferric chloride and raw waste, the water exited into a mechanically mixed 210ℓ (55 gal) container.

An average of 5,550 mg/l of lime (as calcium hydroxide) was added from 10 percent stock tank solution. After a 5-6 minute mix time, the waste flowed by gravity to a second reaction tank where hydrochloric acid was added for pH adjustment. The acid pump was connected to a pH control system set to hold a pH of 9.0 for the wastewater entering the clarifier. This was chosen as a realistic target pH value that would meet the proposed effluent guideline of 7.5 - 10.0.

Approximately 1175 mg/l of 20°Be' hydrochloric acid was used for pH control. Polymer (∿2 mg/l) was injected in-line prior to entering the flocculator-clarifier. The chemically treated wastewater was flocculated for 7-9 minutes and settled. Bench scale testing had determined that a post-floc formed if this clarified effluent was allowed to stand at pH 9.0 and that it would be eliminated by adjusting the pH to 7.0-7.5. Therefore, the settled effluent was pH adjusted between 7.0-7.5 with hydrochloric acid. This required 800 mg/l of 20°Be' hydrochloric acid. The pH adjusted waste was then fed to the dual-media filter. Resulting sludge flow from the clarifier accounted for approximately 45 percent of the raw water flow.

A PAH analytical data summary for the clarifier effluent composite samples collected during the study is shown in Table 2. The data are compared to the raw waste PAH analytical values. In reviewing Table 2, it can be seen that clarification reduced the influent concentrations of dibenzo(a,h) anthracene (140 µg/1) and benzo(ghi)perylene (310 µg/1) to less than 10 µg/1. Acenaphthene was reduced 67 percent by clarification. Fluoranthene, pyrene, chrysene/benz(a)anthracene, 3,4-benzofluoranthene/benzo(k) fluoranthene and benzo(a)pyrene were all removed in excess of 90 percent by chemical clarification. The clarifier average effluent concentration of benzo(a)pyrene was 20 µg/1. During the study, the clarifier removed about 37 percent of the influent suspended solids (TSS). The average effluent TSS was 82 mg/1.

Filtration

Dual-media filtration was used as a polishing step following clarification of the potline scrubber blowdown. The PAH composite sample analysis summary for the dual-media filter effluent is shown in Table 3. A total of nine composite samples were analyzed for PAH's. Comparing the average filter influent PAH concentrations to the effluent (Table 3), it can be seen that dual-media filtration removed up to 50 percent of the residual PAH's remaining in the wastewater following chemical clarification. Effluent average concentrations of benzo(a)pyrene were reduced to 10 µg/1. However, the more soluble PAH's such as fluoranthene and pyrene were only reduced to average values of 110 µg/1 and 80 µg/1, respectively. Thus, this portion of the test program determined that chemical clarification followed by filtration could not consistently achieve PAH concentrations of less than 10 ug/1 in the treated potline scrubber blowdown and removal of benzo(a)pyrene to 10 ug/1 did not result in removals of all PAH's to 10 ug/1.

TABLE 2. COMPOSITE
SAMPLE DATA SUMMARY
FOR PAH ANALYSIS - RAW WASTE
AND CLARIFIER EFFLUENT

PARAMETER	AVERAGE VALUES[A] RAW WASTE	CLARIFIER EFFLUENT	REMOVAL %
1. Naphthalene	ND	ND	-
2. Acenaphthylene	ND	ND	-
3. Acenaphthene	30	10	67
4. Fluorene	ND	ND	-
5. Phenanthrene/Anthracene	ND	ND	-
6. Fluoranthene	2700	170	94
7. Pyrene	2000	110	95
8. Chrysene/Benz(a)anthracene	2200	40	98
9. 3,4-Benzofluoranthene/ Benzo(k)fluoranthene	790	20	97
10. Benzo(a)pyrene	1100	20	98
11. Dibenzo(a,h)anthracene	140	ND	≥93
12. Indeno(1,2,3-cd)pyrene	ND	ND	-
13. Benzo(gni)perylene	310	ND	≥97

ND = not detected (quantification limit = 10 µg/l).

[A]Average was derived from the following number of analyses:
Raw waste - 9
Clarifier eff - 8

All values reported as µg/l without correction for recovery.
Analysis by Method 625.

TABLE 3. COMPOSITE
SAMPLE DATA SUMMARY
FOR PAH ANALYSIS - CLARIFIER EFFLUENT
AND FILTER EFFLUENT

PARAMETER	AVERAGE VALUES[A] CLARIFIER EFFLUENT	FILTER EFFLUENT	REMOVAL %
1. Naphthalene	ND	ND	-
2. Acenaphthylene	ND	ND	-
3. Acenaphthene	10	10	0
4. Fluorene	ND	ND	-
5. Phenanthrene/Anthracene	ND	ND	-
6. Fluoranthene	170	110	35
7. Pyrene	110	80	27
8. Chrysene/Benz(a)anthracene	40	20	50
9. 3,4-Benzofluoranthene/ Benzo(k)fluoranthene	20	10	50
10. Benzo(a)pyrene	20	10	50
11. Dibenzo(a,h)anthracene	ND	ND	-
12. Indeno(1,2,3-cd)pyrene	ND	ND	-
13. Benzo(ghi)perylene	ND	ND	-

ND = not detected (quantification limit = 10 µg/l).

[A]Average was derived from the following number of analyses:
Clarifier eff - 8
Filter eff - 9

All values reported as µg/l without correction for recovery.

Analysis by Method 625.

In addition to removals of some PAH's, filtration removed significant quantities of residual suspended solids (82 percent). The average effluent suspended solids from the dual-media filter was 15 mg/l and ranged from 5-26 mg/l.

Activated Carbon

The activated carbon columns were evaluated for the additional removal of the PAH's present in the effluent from the filter.

During the nine days of testing, 116,200 liters (30,700 gal) of wastewater were processed through the activated carbon columns. The initial two days of testing were performed at a flow rate of 180 $\ell/min/m^2$ (4.5 gpm/ft^2). This was later reduced to 100 $\ell/min/m^2$ (2.5 gpm/ft^2) to allow for better process control of the upstream flocculator clarifier.

The composite sample analyses for the activated carbon system effluent indicated that all PAH's were reduced to less than 10 ug/l for each of the nine samples analyzed.

Grab samples were collected once per day from nine locations around the activated carbon columns. These locations were the first carbon column feed and the midpoint and effluent of each column. The samples were analyzed for fluoranthene, pyrene and benzo(a)pyrene starting with the influent and carbon column no. 1 midpoint. Analysis of additional sample locations was conducted when effluent concentrations of any of the parameters exceed 10 ug/l. Benzo(a)pyrene was selected as one of the parameters for analysis since this compound was evaluated as an indicator for the removal of all PAH's.

Fluoranthene and pyrene were chosen because they were present in significant concentrations and their solubility is greater than that of benzo(a) pyrene (BAP). Thus, removal of these more soluble PAH's via suspended solids removal technology would not be expected to be as good as that for BAP.

The grab samples were all analyzed by Method 610. This is a chromatographic method applicable to the determination of polynuclear aromatic hydrocarbons. To support the Method 610 (2) analytical findings, selected samples were analyzed by an additional qualitative technique [Method 625 (2)]. Method 625 uses gas chromatograph/mass spectrometer (GC/MS) technology to provide results confirmation.

Based on Method 610 results, carbon column influent concentrations of fluoranthene, pyrene and benzo(a)pyrene averaged 40, 50, and <10 µg/l, respectively. Fluoranthene did not exceed 10 µg/l in the carbon column no. 1 midpoint until the sixth day of testing (see Table 4). The volume of waste processed at this time was approximately 75,300ℓ (19,900 gal). After that time, the percent removal continued to decrease each day over the remainder of the test period. By the seventh test day, removal of fluoranthene averaged only 50 percent at the first column midpoint. All values from the effluent of the first carbon column were less than 10 µg/l, however.

Values for pyrene began to exceed 10 µg/l at the carbon column no. 1 midpoint on the fourth day of testing. At this time, 48,100 liters (12,700 gal) of dual-media filter effluent had been processed. The percent removal continued to decrease each day over the remainder of the test period.

TABLE 4. ACTIVATED CARBON DATA SUMMARY - GRAB SAMPLE ANALYSIS

ACTIVATED CARBON COLUMN GRAB SAMPLE POINTS

Sample Location	No. 1 Inf	No. 1 Mid	No.1 EFF
Parameter/Date			
Fluoranthene, µg/1			
7/23/83	30	ND	ND
7/24/83	30	ND	
7/25/83	30	ND	
7/26/83	40	ND	ND
7/27/83	50	ND	
7/28/83	80	20	ND
7/29/83	60	30	ND
7/30/83	50	30	ND
7/31/83	20	ND	ND
Pyrene, µg/1			
7/23/83	20	ND	ND
7/24/83	20	ND	
7/25/83	20	ND	
7/26/83	60	20	ND
7/27/83	60	ND	
7/28/83	60	30	ND
7/29/83	60	30	ND
7/30/83	70	40	ND
7/31/83	60	30	ND
Benzo(a)Pyrene, µg/1			
7/23/83	ND	ND	ND
7/24/83	ND	ND	
7/25/83	ND	ND	
7/26/83	ND	ND	ND
7/27/83	ND	ND	
7/28/83	ND	ND	ND
7/29/83	ND	ND	ND
7/30/83	ND	ND	ND
7/31/83	ND	ND	ND

Analysis by Method 610
ND = not detected (quantification limit = 10 µg/1).
All values reported as µg/1 without correction for recovery.

Since benzo(a)pyrene was effectively removed to <10 µg/1 by clarification and filtration, all grab samples were found to contain <10 µg/1 of benzo(a) pyrene.

Activated carbon also acted effectively as a post-filtration technology for the removal of suspended solids. An average of 47 percent of the residual suspended solids present in the wastewater was removed by the carbon. Effluent suspended solids exiting the carbon columns averaged 8 mg/1 and ranged from 1-15 mg/1.

BENCH-SCALE RESULTS

Introduction

Bench-scale tests were performed on both the potline scrubber wastewater and the anode bake scrubber wastewater.

The objectives of the bench-scale tests were:

1. Determine the chemical doses and operating conditions for total suspended solids (TSS) and PAH removal. Information was also gathered regarding the removal efficiency of aluminum, nickel, antimony, cyanide, and fluoride; however, no attempt was made to optimize removal of these pollutants.
2. Demonstrate that activated carbon would remove the remaining soluble PAH's.
3. Determine if similar PAH removals could be achieved on the two different wastewaters with solids removal technology and activated carbon treatment.

The bench-scale tests were divided into two parts. One part which was conducted by Rexnord in Milwaukee, WI involved determining the required chemical doses and conditions for suspended solids removal via "jar test" experimentation. The other part which was conducted by IERL-Ci involved determining if the soluble PAH's were removed by activated carbon treatment. These bench-scale tests were performed on both wastewaters to determine if matrix interferences existed.

Solids Removal Test Results

Table 5 provides the raw wastewater characteristics determined for the two scrubber wastewaters during the bench-scale testing and also provides the average determinations which were measured during the pilot-scale operation based on 24-hour composite samples.

TABLE 5

RAW WASTEWATER CHARACTERISTICS OF THE SCRUBBER WASTEWATERS

Parameter	Potline		Anode Bake
	Pilot-Scale Composite Avg.	Bench-Scale	Bench-Scale
pH (units)	8.0-9.4	8.6	8.9
Suspended solids (mg/l)	130	190	12
Aluminum (mg/l)	24	23	26
Nickel (mg/l)	1.0	0.8	0.35
Antimony (mg/l)	4.68	< 0.25	1.1
Total Cyanide (mg/l)	38.9	5.8	0.22
Fluoride (mg/l)	874	290	2600

In reviewing Table 5, it can be seen that the anode bake scrubber wastewater contained substantially less suspended solids (12 mg/l) than the potline scrubber wastewater (190 mg/l). Laboratory bench tests determined that the required chemical doses for suspended solids removal for the potline scrubber wastewater was 3,000 mg/l of ferric chloride, 5,300 mg/l of lime as $Ca(OH)_2$, 1,410 mg/l of HCl, and 2 mg/l of Percol 728. This achieved a filtered (Whatman 541) effluent suspended solids of 1.0 mg/l. In comparison, the required chemical doses for solids removal for the anode bake scrubber wastewater was 2,000 mg/l of ferric chloride, 4,000 mg/l of lime as $Ca(OH)_2$, 1,820 mg/l of HCl, and 1.5 mg/l of Percol 728. This achieved a filtered effluent suspended solids of 10 mg/l.

Granular Activated Carbon Bench-Scale Results

A bench-scale granular activated carbon (GAC) column was fabricated and used to evaluate the ability of this technology to remove soluble PAH's from the two primary aluminum scrubber wastewaters. This work was performed by IERL-Ci at the Test and Evaluation Facility in Cincinnati, Ohio. The PAH's were determined by Method 610.

Apparatus and Procedure-- The apparatus consisted of a clear polyvinyl chloride tube [length = 61.0 cm (24"), ID = 2.66 cm (1.05")]; a two liter plexiglass head tank; a metering gear pump; and polyethylene tubing.

Calgon Filtersorb 300 (8 x 30 mesh) was mixed with deionized water to form a slurry and placed in the column to a depth of 30.5 cm (12"). The column was backwashed to remove the fines and kept wet with deionized water.

Raw wastewater was filtered through a 0.45 μ filter and stored at 4°C. The head tank was filled with filtered wastewater and the gear pump was adjusted to a flow rate of 2.8 ml/min (1.07 gal/day) for a 60 minute empty bed contact time (EBCT). For each eight hour period of operation an effluent sample was collected, measured, labelled and stored at 4°C. Also, at this time the head tank was refilled with additional filtered wastewater.

Potline Scrubber Wastewater--Typical PAH concentrations in the raw potline scrubber wastewater which were determined from earlier samples were 790 ug/l, 2,400 ug/l and 2,500 ug/l for benzo(a)pyrene, pyrene and fluoranthene, respectively. Filtration of the raw sample used for the carbon column feed through the 0.45 μ filter resulted in a soluble PAH concentration of 78 ug/l, and 200 ug/l for benzo(a)pyrene, pyrene and fluoranthene, respectively. Thirty-seven liters (9.7 gal) of wastewater was treated and less than 10 ug/l of pyrene and fluoranthene were found in the effluent. The effluent concentration of benzo(a)pyrene varied from 10 ug/l to 20 ug/l.

Anode Bake Scrubber Wastewater--The raw wastewater was found to contain 330 ug/l of benzo(a)pyrene, 970 ug/l of pyrene, and 980 ug/l of fluoranthene. All three of these PAH's were reduced to less than 10 ug/l by filtering through the 0.45 μ filter. As expected, all PAH concentrations in the carbon column effluent were less than 10 ug/l with one exception where 20 ug/l of benzo(a)pyrene was found. Sixty-one liters (16 gal) of wastewater were treated in this test.

CONCLUSIONS

Based upon the results of these studies it can be concluded that:

1. Activated carbon is necessary to consistently remove all PAH's to <10 ug/l.
2. Removal of benzo(a)pyrene to <10 ug/l is not an indicator of the removal of all PAH's to <10 ug/l.

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